

Table 24: Table 23 Discriminant analysis using ($Y = 5.9 \log \text{ length} - 9.2 \log \text{ width} - 6.63$) for all $>0.5 \mu\text{m}$ long fibres.

Sample Number	Type	Count	Discriminant >0	Discriminant <0	% asbestos	% non-asbes tos
HSL/82073/95	Death Valley tremolite	336	315	21	93.75	6.25
HSL/82077/95	Jamestown tremolite	304	278	26	91.45	8.55
HSL/84544/96	Gabbro Germany (air)	167	125	42	74.85	25.15
HSL/82539/95	Chrysotile	288	211	77	73.26	26.74
HSL/83059/96	Tremolite (Edenitic) Drumna drochit	323	204	119	63.16	36.84
HSL/82720/95	Tremolite in dolomite	154	90	64	58.44	41.56
HSL/82761/95*	Tremolite in dolomite	298	174	124	58.39	41.61
HSL/82323/95	NY Talc	342	183	159	53.51	46.49
HSL/82074/95	Tremolite DT	153	69	84	45.1	54.9
HSL/84545/96	Diabase Germany (air)	105	43	62	40.95	59.05

Wylie et al. (1985) did not recommend the use of linear discriminant function analysis in unknown samples, since the magnitudes of the coefficients are sensitive to slight changes in the populations and may therefore be strongly sample dependent. However, the discriminant function was applied to some of the TEM size distribution measurements to determine whether it might apply more widely. For $>5 \mu\text{m}$ long fibres (table 23) the results were well discriminated for asbestos samples with about 89 % of the individual asbestos fibres being classified as asbestos. Similarly for non-asbestos samples only about 22% of non-asbestos mineral fragments were classified as asbestos. The dolomites and New York Talc were in-between with values around 30 - 40%. This showed that the discriminant function was applicable to a wide range of samples and TEM data and may be a useful way to separate samples containing mixed populations of asbestos and cleavage fragments. However, if the fibres are intermediate the results may be difficult to interpret.

The same discriminant function when applied to all fibres with lengths $>0.5 \mu\text{m}$ was less able to discriminate between asbestos and non-asbestos samples with chrysotile giving low values about 70% while a sample which was predominantly mineral fragment HSL/83059/96 Tremolite (Edenitic) Drumnadrochit gave 63% asbestos.

The distinction between asbestos particles and mineral fragments emerges most clearly in their width: virtually no cleavage fragments are $< 0.25 \mu\text{m}$ in width and almost none are $< 0.5 \mu\text{m}$ (Figure 7; Snyder et al., 1987; Kelse & Thompson, 1989). In examining a single fibre < 0.25 or $0.5 \mu\text{m}$ wide, or a small population of such narrow particles, it is reasonable to conclude that they are asbestos. Unfortunately, no conclusion on a fibre-by-fibre basis can be drawn for particles $> 0.5 \mu\text{m}$ wide unless their aspect ratio is $< 3:1$ in which case they lie outside the conventional definition of asbestos fibres and would be taken to be cleavage fragments.

7.8 Discrimination by PLM microscopy

7.8.1 Extinction angle

Extinction angle is perhaps the most important simple observation which can be used to discriminate between amphibole asbestos fibres and cleavage fragments. Due to the random orientation of the fibrils around the c-axis of asbestos fibres, they will show parallel extinction under crossed polars. Non-asbestos monoclinic amphibole fibres (other than anthophyllite) have oblique extinction, except when orientated in the 100 plane. The reliability of this observation for tremolite fibres was tested, by examining the extinction angle of 100 fibres in asbestos and non asbestos samples.

Table 25: Results from PLM measurement of extinction characteristics of various tremolite fibres. (100 fibres analysed)

Extinction	Californian (Jamestown) tremolite	Korean tremolite	Dornie tremolite	HSE reference tremolite
Parallel	43	44	26	53
Oblique	54	52	74	42
Undulating	3	4	0	5

The results in table 25 surprisingly showed that only the HSE reference asbestos (from the Saltworks mine, Death Valley, California) had more than 50% of fibres showing parallel extinction. Tremolite asbestos from both Korea and Jamestown California only has some 43- 44% parallel extinction fibres. A separate analysis of 36 large fibre bundles in the Korean sample gave a higher percentage of parallel extinction (69%). This brings into question how asbestiform tremolite asbestos is and a range of fibre morphologies would seem to be present even in commercially exploited deposits. Tremolite contaminated minerals such as dolomites are unlikely to have a greater percentage of parallel extinction fibres, which suggests that the use of this parameter to discriminate asbestos will not be particularly reliable as a population or individual fibre discriminant. Even the non-asbestos Dornie tremolite had more than a quarter of its fibres giving parallel extinction.

7.9 Summary of discriminant methods

7.9.1 Population Discrimination

The tremolite samples studied were chosen to represent both ends of the range from asbestos to cleavage fragment samples, as well as some intermediate samples, which were either acicular fibres or a mixture of asbestos and mineral fragments. The most successful discrimination between populations of asbestos fibres and cleavage fragments was achieved by measuring the median aspect ratio and median width of the $> 5 \mu\text{m}$ long fibres. Median values of $>20:1$ aspect ratio and median widths $< 0.42 \mu\text{m}$ were characteristic of asbestos and median aspect ratios of $<10:1$ and median widths of $>1.0 \mu\text{m}$ were characteristic of non-asbestos fibre populations. However, no clear width discrimination was found for populations of all fibres $> 0.5 \mu\text{m}$ long but median aspect ratios of $>8:1$ were indicative of asbestos populations.

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Use of an index of fibrosity based on the median aspect ratio raised to the power of the GSD was a useful way to classify a population of $> 5 \mu\text{m}$ long fibres as being asbestos if an index of 200 was exceeded. Although this method may not work well for the wider asbestos populations (e.g. amosite and anthophyllite). For all fibre lengths values of above 50 were indicative of asbestos but the discrimination was much less distinct. A similar attempt to do this with fibre widths was not so successful. Linear regression analysis did not seem to discriminate unambiguously between the asbestos and non-asbestos populations and an arbitrary cut-off has to be selected.

7.9.2 *Individual Fibre Discrimination*

Discriminant function analysis can be used to make a fibre-by-fibre judgements which may have error rates as low as 10-20% for single component samples of asbestos or non-asbestos fibres $> 5 \mu\text{m}$ long, but does less well for all fibre sizes. The effect for samples containing intermediate fibres will depend on the fine adjustment of the values used in the discriminant function..

For tremolite contaminated samples, parallel extinction under crossed polars in a PLM, does not appear to be a reliable discriminant to determine whether the larger ($> 1 \mu\text{m}$ wide and $> 5 \mu\text{m}$ long) fibres are asbestos. Only about half of the asbestos fibres are likely to be counted. However, the inclusion of non-asbestos cleavage fragments which have the (100) plane in line with the illumination and give parallel extinction will partly offset the underestimate.

8. SUMMARY AND CONCLUSIONS

Many of the issues researched and discussed in this report are not new. In the US there was an intense debate between mineral producers and regulators for some 20 years between 1972 -92 which ended when OSHA reversed its previous stance and regulated non-asbestiform mineral fibres as a nuisance dust. Even this ruling has not ended the controversy, as OSHA has been unable to give a method by which asbestos and non-asbestos fibres can be distinguished. It seems that if a producer says that the fibres in their products are a non-asbestos form of amphibole, it is assumed that there is no additional hazard or risk associated with these fibres to the work force or users. With the much greater experimental knowledge and database that has been amassed on the toxicity of non-asbestos fibres since 1992, this regulatory approach is now outdated. Asbestos was the first heavily used industrial fibre and because of the high incidence of respiratory diseases, its use has been regulated separately from 1931. Current knowledge suggests that all fibres have a potential physical toxicity if they are small and durable enough to penetrate and remain in the thoracic regions of the lung for a substantial periods of time. Therefore regulators should be concerned with all durable fibres. The toxicity of any one fibre population being a function of the dose of long fine fibres that is likely to be delivered to the lung.

The current situation is that there is a method for asbestos. Asbestos has many definitions applied to it but is not well - defined in UK or EU legislation. This can give rise to considerable confusion when attempting to assess hazard. There appears to be two main situations where the current definitions of asbestos will produce problems. Firstly, in rocks or mineral deposits containing asbestos fibres which are regarded as contaminants; unless the fibres show the classical commercial and physical properties they can be classified as byssolites or non-asbestiform mineral fibres and deemed to fall outside any asbestos regulations. Secondly, when the current definitions of asbestos fibres in regulatory and International Standard methods for asbestos risk analysis are applied to the mineral production and user industries, a significant proportion of cleavage fragments will be included in the count. Therefore even if a regulatory agency wishes to discriminate between a population of asbestos and elongated cleavage fragments, the current definition for a fibre makes this difficult to do, particularly if a decision is to be made on a fibre by fibre basis.

For hazard assessment the situation is made even more difficult as we need to take into account the potential to produce fibres. This potential has to be realised in the analysis. The method chosen for the EC method is repeated 1 minute grinding in a pestle and mortar of the large fraction 'which does not pass through a 106 µm mesh sieve. However, grinding separates large asbestos fibres into thinner fibrils, so only the larger non-asbestiform fibres are likely to be visible or identifiable by light microscopy. This is of particular concern if a mass percentage determination is used for labelling materials as carcinogenic. Fibre mass is heavily influenced by a small number of large fibres and the counting and analysis of these fibres is the most important factor in the precision of the mass analysis. This suggests that low

magnification (X50-X100) scanning of larger areas of the filter by PCM/PLM to find and evaluate the largest fibres is the best option to improve the analytical precision. However such an analysis concentrates on the thick non-respirable and probably non-asbestos component of the fibres. This produces an anomaly in that the samples with the greatest hazard in terms of mass may actually manifest the least risk if made airborne and sampled by the European reference method (EU Directive 83/477/EEC).

Hazard would be better estimated if the results from the many animal experiments with fibres were applied. Namely, that respirable, durable fibres of the same length or greater than the macrophages are the most toxic and the most difficult to remove from the lung. Therefore the PCM/PLM and any electron microscopic hazard assessment should also record the numbers of >5 , >10 , >15 and >20 μm long respirable fibres produced by the standard challenge (per unit mass) to allow a more precise assessment of the materials toxicity to be made. These data will require no additional work to collect, if a mass analysis is being carried out.

The difficulty in defining and determining whether a fibre is asbestos or not led to an extensive investigation of the shape, mineral and chemical characteristics of asbestos and non-asbestos fibres. The main findings were that accurate TEM length, width and aspect ratio measurements do allow populations to be characterised and estimates of the asbestos and non-asbestos component to be made. This discrimination is much more effective when applied to >5 μm long fibres and samples containing mainly asbestos or mainly mineral fragments. In many mineral samples the fibre growth may produce intermediate shapes or have mixed populations making individual fibre judgements very difficult. For the samples analysed in this work asbestos fibres populations generally have median aspect ratio $>20:1$ and median fibre widths of <0.4 μm and non asbestos fibres and elongate cleavage fragments have median aspect ratios $<10:1$ and median fibre widths >1.0 μm .

Discriminant function analysis did allow individual fibres to be classified as asbestos or non-asbestos but error rates of at least 10-20% can be expected in simple all asbestos and all non-asbestos populations.

As the fibres and mineral fragments are formed from the same mineral any chemical or crystallographic difference are small or subtle and do not offer a simple practical route. However, energy dispersive analysis of individual fibres showed that the minor substitution of Al for Si, is associated with some of the more acicular and prismatic fibres, which do not appear to have the characteristic fibrillar structure of asbestos. More attention could be paid to this aspect in respect to TEM mass analysis to exclude large columnar fibres, particularly as the samples have already been ground to split and release asbestos fibres/fibrils. However, relative small difference in chemistry

The special characteristic that should identify large asbestos fibres is that the fibres are composed of fibrils, which due to the special conditions of formation have produced many parallel oriented fibres along the c-axis. This formation explains the high tensile strength and flexibility properties normally associated with commercially exploited asbestos deposits. Inspection of the ends of large fibres by scanning electron microscopy allows an estimate of how asbestiform the large fibres are due to the presence of many fibrils. SEM also has a 3D image which allows the cleavage faces of mineral fragments to be more clearly seen. However, only a proportion of the large tremolites from a recognised asbestos deposits will show this characteristic structure suggesting that the 'ideal' growth conditions are rarely reached even in commercial deposits of tremolite asbestos. This was further shown when PLM extinction angles were measured on a number of tremolite asbestos samples but <50% of the fibres gave parallel extinction. A lack of a fine fibrillar structure and a tendency towards larger acicular crystals is the most likely explanation.

In some ways PCM/PLM observations of >5 µm long fibres can approximately size the fibres and determine whether the fibre consists of fibrils due to its morphology and or extinction angle. However, it cannot do this as accurately or consistently well as the electron microscopy methods, and will with difficult samples containing mineral fragments, consistently overestimate the asbestos content. This is acceptable if the PCM/PLM is used as a screening method and it is understood that further analysis may be required.

There remains a strong concern that if a population of asbestos fibres is present, even if it is <0.1 % mass, after grinding and if made airborne it could still present a considerable airborne asbestos exposure.

If the EU uses only a mass standard to classify asbestos hazard this will give insufficient information to make informed decisions as to the risk. In some instances the product which contain only large non-respirable cleavage fragments will be labelled as carcinogenic while a substance contaminated with many fine asbestos fibres will not be labelled. Yet when made airborne the unlabelled substance will present a far greater risk.

Both mass and fibre number can be simply measured in a single analysis and should be used to determine the hazard.

9. FURTHER WORK

More field studies are necessary to determine the false positive and negative rates of the PCM/PLM method on real samples before hazard labelling based on this method alone takes place.

The current EU stance on 'asbestos' to the exclusion of other amphibole fibres needs careful appraisal. All the evidence in recent years shows that durable fibres have a physical toxicity. Minor chemical substitutions, a mixed-population of mineral fragments and asbestos fibres or the degree of fibril growth within the larger fibres will be unlikely to reduce the toxicity of the long respirable fibres. Therefore potential carcinogenic fibres may be overlooked if we attempt to rigidly apply the criteria required for asbestos.

A laboratory study to further investigate the relationship between mass and fibre number for contaminated substances needs to be carried out to determine what an appropriate mass or fibre number standard should be.

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